Dry-Grind Process for Fuel Ethanol by Continuous Fermentation and Stripping

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Conversion of a high-solids saccharified corn mash to ethanol by continuous fermentation and stripping was successfully demonstrated in a pilot plant consuming 25 kg of corn per day. A mathematical model based on previous pilot plant results accurately predicts the specific growth rate obtained from these latest results. This model was incorporated into a simulation of a complete dry-grind corn-to-ethanol plant, and the cost of ethanol production was compared with that of a conventional process. The results indicate a savings of $0.03 per gallon of ethanol produced by the stripping process. The savings with stripping result from the capacity to ferment a more concentrated corn mash so there is less water to remove downstream.

Introduction

The Agricultural Research Service, at the Eastern Regional Research Center, has supported the study of process improvements and alternatives to reduce the cost of ethanol production from corn. One approach has been to improve upon existing fermentation processes by combining the fermentation and ethanol separation steps, normally carried out sequentially. Specifically, a new process design, continuous fermentation and stripping, was developed for dry-grind corn to fuel ethanol. In this design, higher solids concentrations are achieved while the otherwise accompanying higher ethanol concentrations are avoided.

The idea that separating ethanol directly from the fermentor can reduce the toxic effect on yeast of its high concentration is not recent. Research on vacuum fermentation was carried out more than 20 years ago (*1*). More recently, extractive fermentation has been developed (*2*). The Biostil process (Chematur Engineering AB, Karlskoga, Sweden) is a commercially available example of fermentation combined with ethanol separation. These processes share a common feature: because ethanol is removed during the fermentation, a more concentrated feed stream can be converted to ethanol. When such a process is applied to whole, dry-milled corn, the solids concentration of the mash going into the fermentor can be increased. There is less water to remove downstream, and the costs for stillage dewatering equipment (centrifuge and evaporator) are reduced.

Utilizing the inert, noncondensable byproduct CO_2 gas to strip ethanol from the fermentor was proposed 15 years ago (*3*). In recent years, several research groups have studied ethanol fermentation with stripping (*4*–*6*). Stripping can be done in an airlift fermentor, where the CO_2 gas provides mixing as well as stripping. However, in continuous fermentation and stripping, the quantity of gas needed for stripping may be much larger than for mixing. To avoid the high cost and energy consumption of a large compressor, we proposed recycling the fermenting mash through a stripping column. A less expensive blower or fan could then provide the required gas flow at much lower pressure (*7*).

During pilot plant testing, it was found that growth of yeast cells attached to the packing in the stripping column could block the flow of stripping gas. Twice weekly washing of the column controlled the fouling and made it possible to operate stable continuous fermentation and stripping runs lasting up to 6 months. A concentrated feed containing glucose (560 g/L) and corn steepwater was completely converted. Detailed results from this work have been published, including a mathematical model to fit the data (*7*–*10*). Now the results from the continuous fermentation and stripping of a saccharified corn mash containing high levels of suspended solids are presented. Estimated operating costs for continuous fermentation and stripping are compared with a state-of-the-art dry-grind process based on continuous cascade fermentors.

Materials and Methods

Yeast. The yeast ATCC 4126, *Saccharomyces cerevisiae*, reported to have a high-temperature tolerance and used in the vacuum fermentation work (*1*), was obtained from the American Type Culture Collection, Rockville, MD. It was maintained on YM Agar slants (YM Agar, YM Broth from Difco, Detroit, MI), sealed and kept in the refrigerator for up to 1 year. The contents of two slants were resuspended in 4 L of YM Broth, grown in four 2-L shake flasks at 25–30 °C overnight and used to inoculate the fermentor.

Saccharified Corn Mash. Fine cracked corn (13% moisture) was purchased in 23 kg (50 lb) bags from a local feed mill and stored for up to 1 week at room temperature. The empty bags, woven from plastic strips, were used to collect the spent grains following fermentation. The beer that passed through the bag was collected and heated to evaporate at least half of the ethanol, providing a material similar to so-called "backset", typically used at 15% of the final mash volume. The backset

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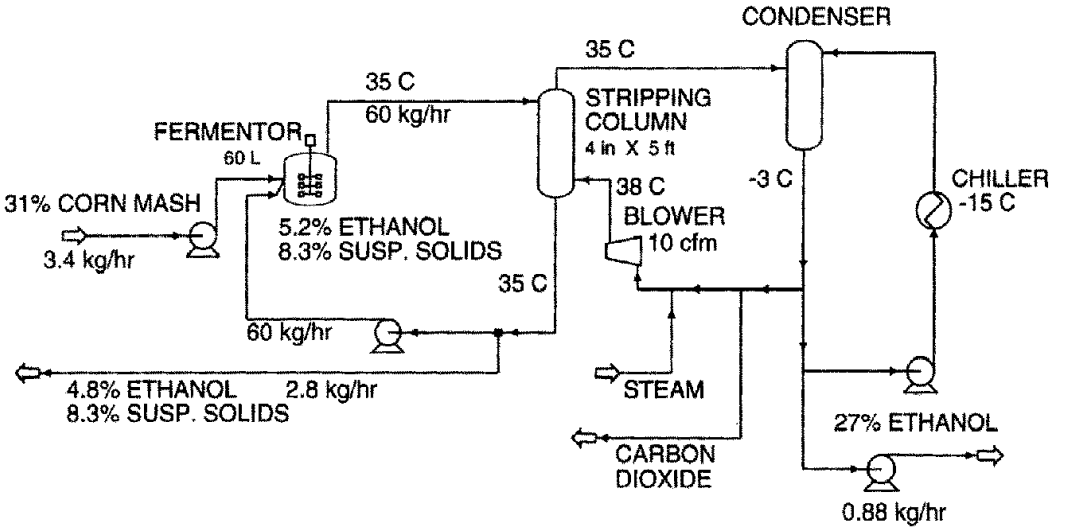

Figure 1. Continuous fermentation and stripping pilot plant. All concentrations are in weight percent.

(47 kg) was added to warm tap water (136 L) in an insulated, 388 L (100 gallon), loosely covered, stainless steel tank equipped with cooling coils, a steam sparger, and an air-driven stirrer. Ammonium chloride (150 g) was added, the pH was adjusted to 6.0 with ammonium hydroxide, and steam was added with stirring. When the temperature reached 90 °C, 59 kg (130 lb) of fine cracked corn was added, and the stirrer speed was increased. After cooking for 10 min, 160 mL of thermostable α-amylase (Spezyme Delta AA from Genencor International, Rochester, NY) was added by means of a peristaltic pump at 0.9 mL/min.

After cooking with enzyme for 30 min, the mash was recycled through 1.3 cm (1/2 in.) stainless steel tubing using a brass gear pump (1/2 HP). The α-amylase addition point was located at the gear pump discharge to mix the enzyme with the recycle stream just before it returned to the tank. The temperature was maintained at 85–90 °C by adjusting the rate of steam addition while an additional 55 kg (120 lb) of cracked corn (250 lb total) was gradually added over the next 2 h or more. Occasionally it was necessary to remove uncooked corn that clogged the gear pump suction line by backflushing with hot water. After all of the corn was cooked and liquefied, the pH was adjusted to 4.4 with sulfuric acid, and the mash was cooled below 70 °C before 150 mL of glucoamylase (Genencor Optidex L-300) was added. The mash was continuously stirred, and the temperature was maintained at 55 °C for approximately 4 days while the mash was fed to the fermentor. The gear pump was left on for the first 2 days to reduce the particle size of suspended solids and prevent clogging of transfer lines. The last 100 L, approximately, of feed was transferred to an auxiliary feed tank, to maintain continuous operation of the fermentor and stripping column, while the main tank was cleaned and the next batch of saccharified corn mash was prepared.

Equipment. The arrangement of equipment is indicated in the flow diagram in Figure 1. The fermentor was a 60 L jacketed, stainless steel pressure vessel. The feed rate was manually set using a timer that pulsed a peristaltic pump for approximately 3 min every 10 min. The pH was controlled between 3.5 and 3.8 by automatic addition of 10% aqueous ammonia (ammonium hydroxide). Air was supplied to the sparger at approximately 1 L/min. The fermentor was stirred at 250 rpm, and the temperature was maintained at 35 °C. The working volume, including recycle holdup was 61 L. The fermenting mash was recycled with a peristaltic pump (model 604 S/R, Watson-Marlowe, Wilmington, MA), which pumped approximately 60 kg/h from the bottom of the stripping column to the bottom of the fermentor. The recycle pump provided sufficient pressure to force the mash out through the fermentor exhaust port with the produced CO_2 and through a transfer line to the top of the stripping column. Transfer lines for feeding and recycling the mash consisted of thick-walled, insulated, rubber tubing with 1 cm (3/8 in.) or larger inside diameter.

Recycled mash flowed through a temperature-controlled heater installed in the top plate of the stripping column and into a 10 cm (4 in) × 1.5 m insulated glass column packed with 10 disc-and-donut trays (Figure 2). Accumulated CO_2 stripping gas was recirculated at approximately 300 L/min (10 cfm) through a 7.6 cm (3 in.) × 1.2 m stainless steel condenser using a plastic, centrifugal blower having a 38 cm (15 in) diameter impeller with 10 cm (4 in) intake and outlet. The blower speed was constant at 1350 rpm. The stripping column, condenser, and blower were connected using 10 cm glass and stainless steel ducts, pipes, elbows, and tees. The pressure was less than 3 cm of water throughout the gas loop. Spent grains and beer exited by gravity from the bottom of the stripping column through an overflow pipe along with exhaust gas. Two times per week during continuous operation, the blower and recycle pumps were turned off and a centrifugal pump was used to recirculate 6–8 L of 30% ethanol (condensate) through the stripping column at 8–10 L/min for at least 10 min to wash attached yeast cells from the packing.

The condenser was packed with 2.5 cm (1 in.) stainless steel Intalox (Norton, Akron, OH). Condensate from the bottom of the condenser was recycled with a stainless steel gear pump (Micropump, Concord, CA) through a 0.09 m^2 (1 ft^2) stainless steel heat exchanger and back to the top of the condenser. The heat exchanger was cooled by methanol at −15 to −20 °C supplied by a 1 HP chiller (FTS Systems, Stone Ridge, NY). The heat exchanger, condenser, coolant, and condensate recycle lines were insulated. A positive displacement pump was used to withdraw produced condensate from the bottom of the condenser. The refractive index of the condensate was continuously monitored using a fiber optic probe and instrument (Photonetics, Wakefield, MA).

The cold gas leaving the condenser was reheated and humidified by direct steam injection. Steam condensate

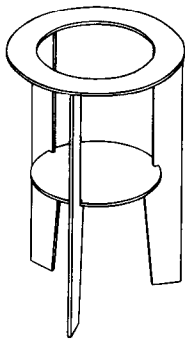

Figure 2. Disc-and-donut packing; welded stainless steel; diameter 10 cm (4 in); height 15 cm (6 in); 10 pieces were stacked in the stripping column.

drained by gravity through a U-tube trap to prevent gas from entering or leaving. The temperature of the gas entering the bottom of the stripping column was controlled by using a mechanical temperature controller to adjust an air-operated valve on the steam supply line. The temperature controls on the column and fermentor provided for temperatures constant to within 1 °C when the room temperature changed by 5 °C or more. The temperatures of the gas and liquid entering and leaving the stripping column and leaving the condenser were measured with thermistors and recorded with a data logger (J-Kem Scientific, St. Louis, MO). On-line measurements of the pH, temperature, and pressure in the fermentor and refractive index of the condensate were also logged.

Analyses. The overflow, condensate, and feed flow rates were measured at least once every day. For glucose, ethanol, and cell count measurements, a sample (approximately 100 g) of the overflow was taken at least once a day, into a 300 mL beaker containing approximately 0.1 g of sodium azide to stop sample fermentation. The overflow usually flowed only when the feed pump was pulsed on. Because the sample was taken during the first minute of flow, and because the feed was pumped directly to the fermentor and the sample was taken from the stripping column, the sample was taken at the lowest glucose concentration during the 10 min feeding cycle. The highest glucose concentration during the cycle was approximately 1 g/L higher.

Cells were counted using a microscope and hemacytometer after diluting 1/100 with distilled water. Glucose and ethanol were measured on the clear supernatant following centrifugation. The previously tared centrifuge tube containing the wet pellet was dried at 100 °C overnight to determine the dry weight of suspended solids (yeast and spent grains). The glucose concentration was measured with a YSI (Yellow Springs Instrument Co., Yellow Springs, OH) glucose analyzer, and the feed rate was manually adjusted to maintain a small (less than 5 g/L) but measurable (greater than 0.2 g/L) glucose concentration. Ethanol concentrations in the overflow and in the condensate (also sampled daily) were measured by HPLC using an HP 1050 (Hewlett-Packard, San Jose, CA) with a Fast Acid Analysis Column, 100 mm × 7.8 mm (Bio Rad, Hercules, CA) and a differential refractometer (Spectra Physics, Riviera, FL). The column temperature was 70 °C, and the mobile phase was 0.001 M H_2SO_4 at 0.7 mL/min.

Total solids of the saccharified mash and moisture content of the fine cracked corn were determined by drying in a vacuum oven at 70 °C. To measure the starch content of fine cracked corn, the corn was ground in a Wiley mill fitted with a no. 20 mesh screen, and 200–300 mg of the ground corn was mixed with 25 mL of deionized water and gently boiled, with stirring, for 3–5 min. The mixture was autoclaved for 1 h at 135 °C and incubated with amyloglucosidase (Starch Assay Kit, reagent S-9144, Sigma, St. Louis, MO) at 60 °C for 15 min. Glucose was determined using the YSI analyzer, and starch was calculated by multiplying total glucose by 0.925.

Computer simulation and cost analysis were performed using ASPEN PLUS (Aspen Technology, Cambridge, MA). Data from 12 consecutive days of continuous steady-state operation, including data from 3 consecutive batches of feed, were averaged. The averaged pilot plant data were used as inputs to a simulation of the process to determine the stripping gas flow rate and to gauge the effectiveness of the disc-and-donut packing. The glucose conversion, ethanol and cell yield, and specific growth rate were also calculated from the simulation results. The simulation included small losses of ethanol with the steam condensate and CO_2 exhaust, which were not measured. A separate, more complex simulation, including sizing and costing of all equipment, was constructed to represent a full scale (15 million gallons per year) state-of-the-art dry-grind ethanol plant. Then a model for continuous fermentation and stripping based on pilot plant results was substituted for the continuous cascade fermentors in this simulation, the heat integration pathways were modified, and the process flowsheet was reoptimized.

Results

Data from only the last 12 days of steady-state operation are reported here, but continuous operation of the pilot plant on saccharified corn mash proceeded for 96 days. There was no contamination of the fermentor during or at the end of this operating period. Except for the first few days, the condensed ethanol production rate was constant at 0.8–0.9 kg/h during the entire run. At startup, the saccharified mash was lower in total solids (20–25 wt %) and the stripping column contained the random packing used in previous runs on feed without suspended solids (*10*). Within the first few days, the column plugged with corn fiber. Although washing restored some flow of stripping gas, steady state could not be achieved. The random packing was then replaced with 10 disc-and-donut trays, and the solids concentration of the feed was gradually increased. As the feed concentration increased, the condensed ethanol concentration increased from approximately 150 to 250 g/L. Splashing of the mash onto surfaces at the top and bottom of the column that were not washed with ethanol allowed fungus to grow there. However, where the column was washed, the washing procedure was completely effective; there was no fouling of the column walls or trays.

The saccharified mash (average of last 3 batches) had specific gravity 1.115 at 50 °C and contained 33.9 wt % total solids. Subtracting the backset (0.15 × 5.0 wt % total solids) and water of hydrolysis (0.1 × 22.9 wt % glucose), the mash contained 30.9 wt % corn dry solids. Although some remaining starch was detectable by iodine test, the conversion of starch to glucose was essentially complete, because the measured amount of glucose in the saccharified mash was equivalent, within experimental error, to 100% of the theoretical maximum conversion of the measured amount of starch (66.8% dry basis) present in the original corn. There was no starch detectable by iodine test in the spent grains or beer.

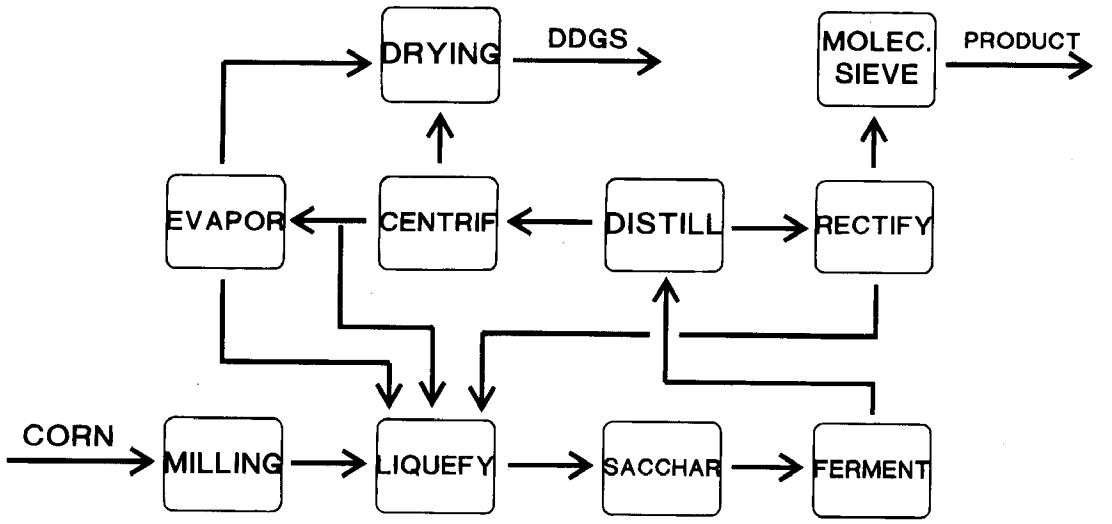

Figure 3. State-of-the-art dry-grind process for fuel ethanol.

The averages of measured values that were used as input to the pilot plant simulation included feed flow rate (3.36 kg/h); condensate flow rate (0.879 kg/h); ethanol in condensate (257 g/L); ethanol in overflow (52.0 g/L); glucose in overflow (0.12 g/L); cell count in overflow (5.8×10^8 cells/mL); suspended solids (including yeast) dry weight in overflow (8.3 wt %); temperature of gas entering bottom of stripping column (37.9 °C); temperature of gas leaving top of stripping column (35.3 °C); and temperature of gas leaving condenser (−3.4 °C). Some of the measured and result variables are also shown in Figure 1. Other important simulation results were the ethanol concentration in the fermentor (55.8 g/L), the ethanol yield (0.48 g/g glucose or 94% of theoretical), and the stripping gas flow rate, 27.0 kg/h. The overflow rate was measured but not used as a simulation input. The agreement between the measured overflow rate (2.70 kg/h) and the simulation result (2.79 kg/h) is an indication of the validity of the simulation and the internal consistency of the measured data.

In the simulation, the stripping column was handled as a single equilibrium stage (flash block), with variable bypass of the gas stream. That is, a fraction of the stripping gas was assumed to channel through the stripping column without contacting the liquid, while the remainder was assumed to be in equilibrium with the liquid flowing from the column. The bypass fraction was automatically adjusted within the simulation until the overflow and condensate ethanol concentrations agreed with the measured data. The resulting bypass fraction was 0.45. In other words, the stripping column with 10 disc-and-donut trays can be described as equivalent to 0.65 of a theoretical equilibrium stage. Previous analysis of random packing in the same column indicated performance varied from 0.3 to 2.0 theoretical stages (*10*). Wall effects are large at this scale, so the height of a theoretical stage for scale-up cannot be accurately estimated.

The specific growth rate (μ) was calculated from the simulation results to be 0.045 h^{-1}. The concentration factor previously defined as the glucose concentration in the feed multiplied by the ratio of feed rate to overflow rate (*10*) calculated from these latest results is considerably less than the lowest value among data previously used for mathematical modeling. Therefore, to compare the present result with the previous results, it was necessary to fit the previous data to a new model that does not include the concentration factor. The new model is

$$\mu = 0.07647\left(\frac{S}{S + 0.0892}\right)\left(1 - \frac{P}{65.7}\right)^{0.2385}\left(1 - \frac{T - 34}{12.56}\right)$$

where S is substrate (glucose) concentration (g/L) in the fermentor, P is product (ethanol) concentration (g/L) in the fermentor, and T is the stripping temperature (C). Here, T is a measured variable and P is a simulation result, but S is unknown in the present situation because the feed was pulsed. Although the average of measured values was only 0.12 g/L, this was the lowest value during the feeding cycle. For most of the cycle, the value of S was large compared to 0.0892 g/L, so the term containing S is approximately equal to 1. Eliminating the substrate term and setting $P = 55.8$ and $T = 35.3$, the above model predicts $\mu = 0.0437$ h^{-1}, in close agreement with the latest result (0.045). It can be concluded that the above model, based on data obtained using a glucose and corn steepwater feed, is also applicable to continuous fermentation of saccharified corn mash, at least at ethanol concentrations near 55.8 g/L.

Based on the previous results (*10*) and eliminating the concentration factor as described above, the new model for cell yield is

$$Y_{x/s} = 0.0641\left(1 - \frac{P}{65.7}\right)^{0.2385}$$

This model predicts that at 55.8 g/L ethanol the cell yield is 0.0408 g/g glucose. Applying this model to the present situation, the number of cells in a gram of cell dry weight and the cell concentration in the fermentor can be calculated to be 5.0×10^{10} cells/g and 11.7 g/L.

Cost Estimation

The cost of continuous fermentation and stripping was evaluated in comparison with a base-case, state-of-the-art process for ethanol production by corn dry-milling. A leading designer of such plants generously provided us with details of the process shown in Figure 3. Annual production is approximately 15 million gallons per year using continuous cascade fermentors and molecular sieve dehydration of ethanol. The saccharified mash entering the fermentors contains 23 wt % corn (dry solids), and the stream passing from the fermentors to distillation contains 9 wt % ethanol. The corn is cooked with hot evaporator condensate and distillation bottoms, so that little fresh makeup water is required and almost no wastewater is produced. To limit steam consumption and

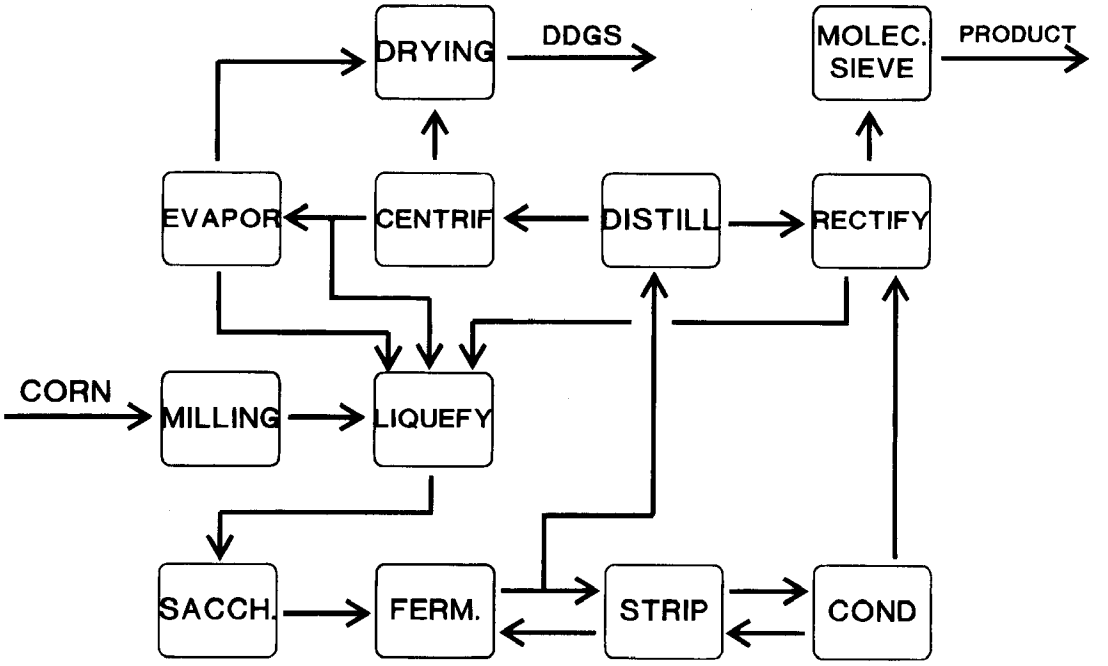

Figure 4. Dry-grind process with continuous fermentation and stripping.

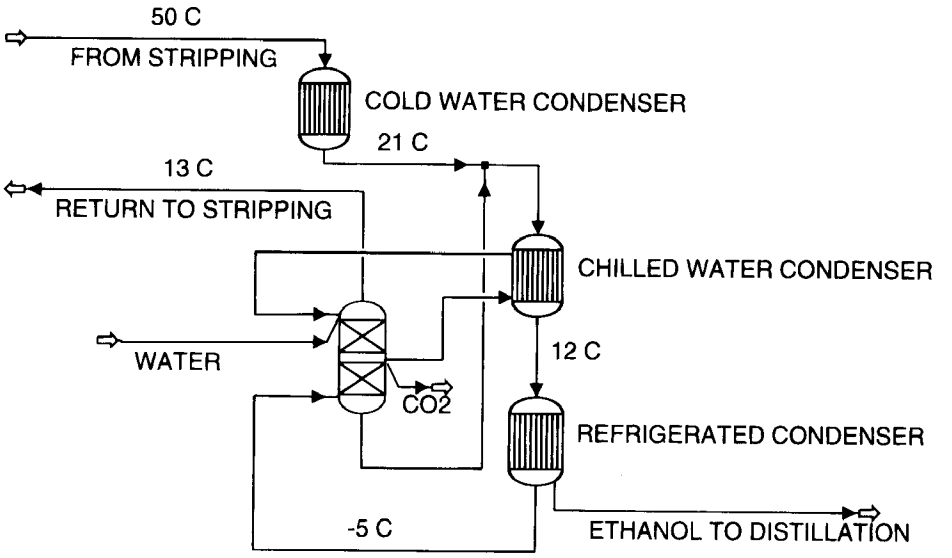

Figure 5. Three-stage condenser with chiller/absorber column.

to provide heat to a six-effect vacuum evaporator, the heat from condensing the distillation overhead vapors required for reflux to the rectifier column is utilized.

A dry-grind process for ethanol production by continuous fermentation and stripping, adapted from the process shown in Figure 3, is shown in Figure 4. A major difference from the conventional plant is that the solids concentration entering the fermentor is increased to 34 wt % corn (dry solids). Although the amount of corn processed is the same, the flow rates of the more concentrated streams are lower. In particular, reduced flow rates to fermentation, distillation, centrifugation, and evaporation lead to smaller sizes and lower equipment costs for these sections. The ethanol concentration leaving the fermentor is decreased to 6 wt %, but only about half of the total ethanol flows to the beer still with this stream. The other half flows directly to the rectifier still from the stripping condenser, at 33 wt %.

Cost estimates based on information from a refrigeration system vendor showed that a single refrigerated condenser, as in our pilot plant, would be too expensive for a commercial process. An alternative is absorption to recover ethanol from the gas phase, either on a solid adsorbent, or into a solvent. However, we designed a cost-

Table 1. Capital Costs Comparison (in dollars) for Nominal 15 Million Gallons per Year Ethanol Plant

processing section	current practice	CO_2 stripping	differences
grain unloading & milling equip	2,270,000	2,270,000	0
saccharification equip	2,167,000	1,820,000	(347,000)
fermentation	3,633,000	2,080,000	(1,553,000)
stripping	0	3,190,000	3,190,000
distillation & dehydration	2,580,000	2,440,000	(140,000)
evaporation & drying	8,240,000	6,000,000	(2,240,000)
product handling	1,080,000	1,080,000	0
utilities	1,190,000	960,000	(230,000)
total, capital costs	21,160,000	19,840,000	(1,320,000)

Table 2. Production Cost Comparison (in dollars) for Nominal 15 Million Gallons per Year Ethanol Plant

plant outputs	current practice	CO_2 stripping	annual differences	savings $/gal
ethanol production (gal/year)	14,694,444	14,754,801	60,357	
DDGS production (tons/year)	53,986	53,673	(313)	
		production costs		
raw materials	14,655,000	14,510,000	(145,000)	0.014
utilities	2,498,000	2,500,000	2,000	0.001
labor, supplies, and plant overheads	3,723,000	3,619,000	(104,000)	0.008
depreciation	2,351,000	2,204,000	(147,000)	0.011
subtotal net operating costs	23,227,000	22,833,000	(394,000)	0.033
less byproduct credits	(6,480,000)	(6,443,000)	37,000	(0.004)
annual ethanol production cost	16,747,000	16,390,000	(357,000)	0.029

effective three-stage condensing system. As shown in Figure 5, it consists of three condensers and a packed chiller/absorber column. Only the third stage condenser requires refrigeration, to cool the gas to −5 °C. Remaining uncondensed ethanol vapor is absorbed by cold water in the bottom section of the packed column. The top section operates like a cooling tower, producing chilled water for the second stage condenser. The first stage condenser uses cold well water, available depending on plant location. The chiller and condenser of the pilot plant are replaced by the three-stage condenser in the full scale simulation.

Detailed cost analyses of both processes were carried out using the Aspen process simulator. Quotations from equipment vendors were used to modify the Aspen default cost models and to create cost models for process equipment not included with Aspen built-in costing. The various factors and assumptions required to size and cost the equipment and then calculate the operating cost were the same for both processes. For example, in both cases the total capital investment was estimated to be 3 times the total equipment purchase cost, and the capital investment was depreciated evenly over 9 years. The operating labor and total cost of corn were the same for both processes. Estimates for the major processing sections and cost categories are shown in Tables 1 and 2. The last column of Table 2 is calculated as the difference between current practice (annual dollars/annual gallons) and stripping (annual dollars/annual gallons). Table 2 shows that the stripping process saves approximately $0.03 of net operating cost per gallon of ethanol produced. Similar cost analyses using different assumptions and compared against different base cases have invariably indicated significant cost savings for the stripping process.

The largest source of cost savings for the stripping process is the capital investment, as shown in Table 1. Both depreciation and maintenance labor and supplies, which contribute combined savings of $0.019 per gallon as shown in Table 2, are calculated as percentages of the total capital investment. These savings result primarily from decreased sizes for the fermentor, beer still, reboiler, centrifuge, and evaporator. Also, a separate scrubber to recover ethanol from the fermentor exhaust is not required with stripping.

The capital costs for utilities shown in Table 1 do not include the steam boiler, cooling tower, or refrigeration plant. These costs are included in the price of utilities and are reflected in utility consumption costs in Table 2. Total utility costs are almost the same for both processes, because in the stripping process costs for refrigeration are offset by lower steam consumption in the beer still reboiler. Also, heat that is removed with cooling water from evaporator condensers in the base case can be utilized in the stripping process to reheat the cold stripping gas to 40 °C and saturate it with water vapor. The elimination of the requirement for purchased yeast, reflected in the raw materials cost in Table 2, is another significant advantage of continuous fermentation and stripping.

The concentration of corn (34% dry solids) in the stripping process is our estimate of the limitations of conventional liquefaction and saccharification equipment. With different equipment, it might be possible to produce a mash containing up to 40% dry corn. It is likely that the stripping process operating on such a mash would show a greater overall cost advantage, despite higher costs for liquefaction and saccharification.

Conclusions

Continuous fermentation and stripping of saccharified corn mash containing 34 wt % total solids was successfully demonstrated in a pilot plant consuming 25 kg of corn per day. There was no contamination of the fermentor during 96 days of continuous operation. Disc-and-donut packing in the stripping column effectively handled the suspended solids, and washing prevented growth of yeast and fungus in the stripping column. Results from 12 consecutive days of steady-state operation were in agreement with a kinetic model derived from previous data. Computer simulation and cost analysis of a base-case state-of-the-art dry-mill plant showed that substitution of continuous fermentation and stripping for continuous cascade fermentors results in an overall cost savings of $0.03 per gallon of ethanol produced. The savings are due primarily to approximately 50% higher solids concentrations, reducing the load on byproduct dewatering equipment and lowering the total capital investment by over $1,000,000. With some modifications, the process may show greater savings at higher solids concentrations.

Acknowledgment

Some of the data for the base-case state-of-the-art dry-grind process were provided by Delta-T Corporation, Williamsburg, VA.

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